

TRANSFORMATIONS OF 4H-1-BENZOPYRAN DERIVATIVES

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Abstract

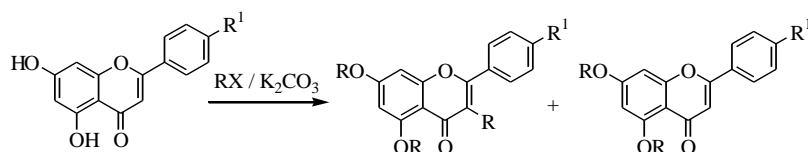
4H-1-Benzopyran derivatives are seen to be transformed in a number of ways to other varieties of newer heterocycles related to them mainly with a view to getting biologically active molecules. A good number of such transformations appearing in the current literature are presented in this Section.

Introduction

Six-membered oxygen containing heterocycles are widespread through both the domains of natural and synthetic compounds. Compounds containing pyran, benzopyran ring systems display interesting biological activities, which have motivated the chemical community towards the studies on their isolation, structure, reactivity and synthesis. A great deal of these materials has been reviewed and the focus of this article is to present an overview on the synthesis and reactivity of the said classes of compounds developed in recent years. The important group of natural benzopyran compounds is the flavonoids which abundantly occur in the plant kingdom. Their intake takes place through foods and they perform many important biological functions. The main objectives of synthesis of chromones and related compounds are not only for the development of more diverse and complex compounds having wide range of biological activities and their structure-activity relationship studies but also for other applications in medicinal chemistry and material science, such as development of various fluorescence probes due to interesting photophysical and photochemical properties of these compounds.

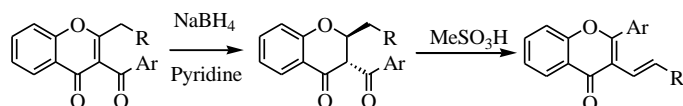
Transformation of 4H-1-benzopyran derivatives

Wang *et al.* developed [1] the synthesis of 3-alkylated flavanoid derivatives. Treatment of B-ring substituted 5,7-dihydroflavones with alkyl halides in the presence of potassium carbonate gave unexpected 3-alkylated flavonoids. A ring opening followed by alkylation and ring closure mechanism was proposed.



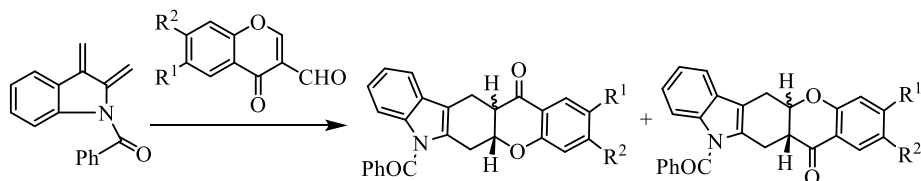
Scheme 1

Clarke *et al.* reported [2] reduction of chromon 4- one with NaBH_4 in pyridine to give the corresponding chroman-4-one in high yield. The reduction is both regio- and chemoselective. Treatment of the chroman-4-ones with MeSO_3H generated the 3-alkenyl-2-arylchromones by a dehydrative rearrangement initiated by retro-Michael cleavage of the pyranone ring.



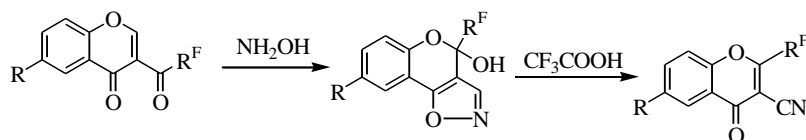
Scheme 2

Terzidis *et al.* explained cycloaddition reactions of chromone-3-carboxaldehydes with indole-*o*-quinodimethane gave [3] a diastereomeric mixture of Diels–Alder cycloadducts in good yields after *in situ* deformylation.



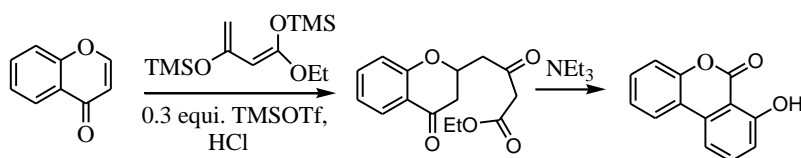
Scheme 3

Sosnovskikh *et al.* reported [4] that the reaction of 3-(polyfluoroacyl)chromones with hydroxylamine proceeded *via* nucleophilic 1,4-addition followed by opening of the pyrone ring and subsequent cyclization to 4-(polyfluoroalkyl)-4*H*-chromeno[3,4-*d*]isoxazol-4-ols in good yields. On treatment with trifluoroacetic acid, the isoxazole ring of this annulated heterocyclic system opened to give 3-cyano-2-(polyfluoroalkyl)chromones. Reaction of 3-(polyfluoroacyl)chromones with hydroxylamine hydrochloride was reported to occur only at the carbonyl carbon atom connected to the R^F group to give the corresponding oximes.



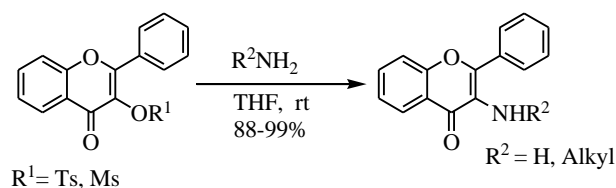
Scheme 4

Appel, *et al.* reported [5] the condensation of 1,3-*bis*-silyl enol ethers with benzopyrylium triflates, generated *in situ* by the reaction of chromones with Me₃SiOTf, afforded functionalized 2,3-dihydrobenzopyrans; treatment of the latter with NEt₃ or BBr₃ resulted in a domino retro-Michael–aldol–lactonization reaction with formation of a variety of 7-hydroxy-6*H*-benzo[*c*]chromen-6-ones.



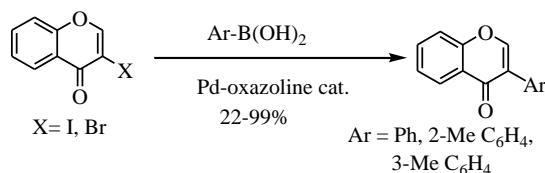
Scheme 5

Takechi *et al.* reported [6] Michael addition followed by elimination of alkoxy group with the formation of an aziridine intermediate which opens up to give the product.



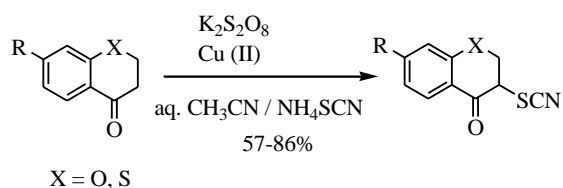
Scheme 6

Eisnor *et al.* described [7] the use of an air stable Pd–oxazoline catalyst as mediator of selective C–C bond formation reactions. This methodology was then applied for the synthesis of a number of biologically active stilbene and isoflavone derivatives.



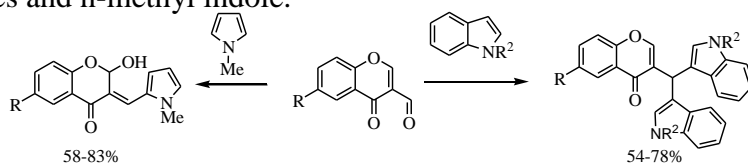
Scheme 7

Kumar *et al.* developed [8] a convenient approach for the direct α -thiocyanation of carbonyl and β -dicarbonyl compounds using ammonium thiocyanate as the thiocyanating agent and potassium peroxydisulfate as the oxidant in the presence of a catalytic amount of copper(II) sulfate in aqueous acetonitrile



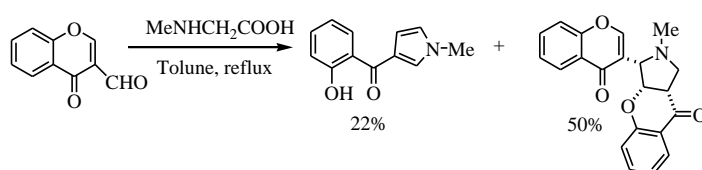
Scheme 8

Sosnovskikh *et al.* synthesised [9] chromon-3-ylbis(indol-3-yl) methanes from chromone-3-aldehyde and indole in good yields. Chromone-3-aldehydes have been synthesised from *E*-2-hydroxy-3-(1-methylpyrrol-2-ylmethylene)chroman-4-ones and *n*-methyl indole.



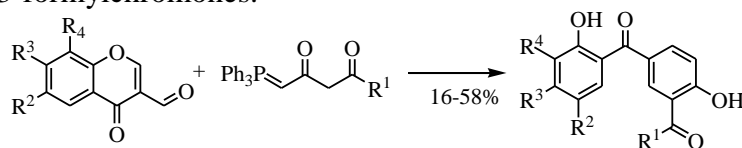
Scheme 9

Andrea *et al.* synthesised [10] azomethine ylides generated from the reaction of chromone-3-carbaldehyde with amino acids undergo 1,5-electrocyclization reactions to afford 3- and 4-(2-hydroxy-benzoyl)pyrroles



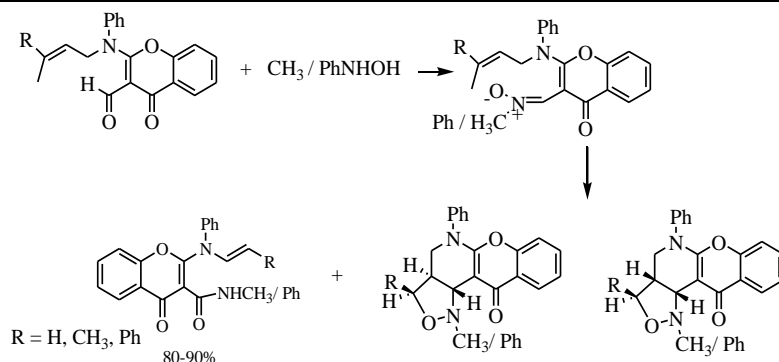
Scheme 10

Holtz *et al.* prepared [11] 4-(2-Hydroxybenzoyl)salicylic esters and amides prepared by domino Michael-Retro-Michael-Wittig reactions of (2,4-dioxobutylidene)triphenyl phosphoranes with 3-formylchromones.



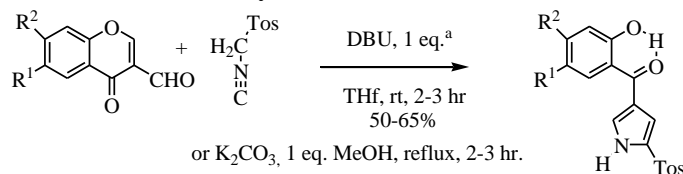
Scheme 11

Singh *et al.* reported [12] Facile intramolecular 1,3-dipolar cycloadditions of *in situ* generated nitrene on heterocyclic systems, reactions of 2-(*N*-allyl/crotyl/cinnamyl-anilino)-3-formylchromones with *N*-phenyl-/methylhydroxylamine under comparable conditions, afford fused isoxazolidines only in low to moderate yields; the corresponding amides derived from rearrangement of *in situ* generated nitrenes are formed as major products.



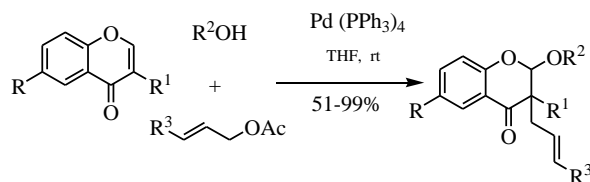
Scheme 12

Terzidis *et al.* reported [13] the reaction of Chromone-3-carboxaldehydes with tosylmethylisocyanide (TOSMIC) in the presence of DBU in THF at room temperature to furnish, after *in situ* deformylation, good yields of 2-tosyl-4-(2-hydroxybenzoyl)pyrroles. When the same reaction is performed with potassium carbonate in methanol under reflux pyrroles are again isolated, but in lower yields (13-35%).



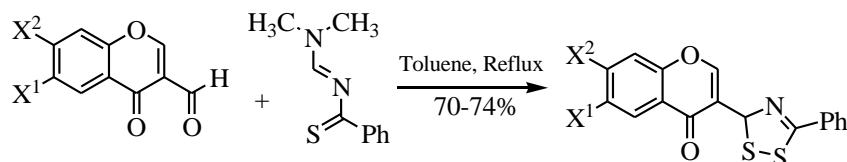
Scheme 13

Patil *et al.* explained [14] palladium catalyzed highly efficient three component coupling (TCC) reactions between chromones, allylic acetates and alcohols, which led to a library of multiply substituted chromones. The reaction proceeded *via* the formation of benzopyrylium cation, generated from the reaction between chromones and allyl acetate, in the presence of palladium catalyst.



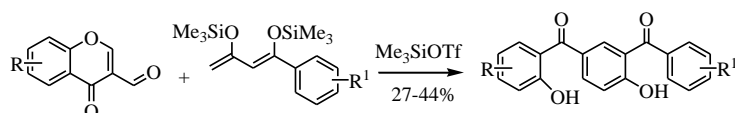
Scheme 14

Raj *et al.* explored [15] the formation of novel substituted 3-(5-phenyl-3H-[1,2,4]dithiazol-3-yl)chromen-4-ones in high yields. Substituted 3-formylchromones react with 2-phenyl-4-dimethylamino-1-thia-3-azabuta-1,3-diene or thio-benzamide by heating their toluene solution in a sealed tube to give the result.



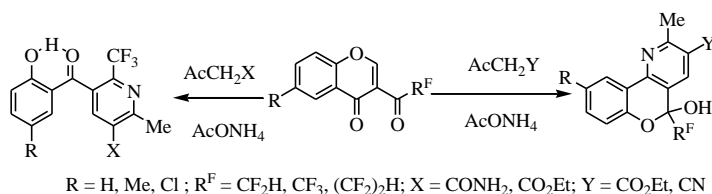
Scheme 15

Yawer *et al.* reported [16] domino reaction of 1-aryl-1,3-bis(silyloxy)buta-1,3-dienes with 3-formylbenzo-pyrylium triflates, generated *in situ* from 3-formylchromones, afforded a variety of 2-benzoyl-4-(2-hydroxybenzoyl)phenols.



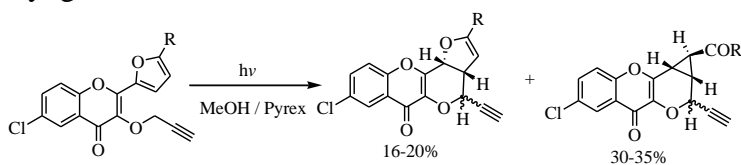
Scheme 16

Sosnovskikh *et al* explained [17] the reactions of 3-(polyfluoroacyl)chromones with acetoacetamide and ethyl acetoacetate in the presence of ammonium acetate proceed at the C-2 atom of the chromone system with pyrone ring-opening and subsequent cyclization to yield 5-salicyloyl-2-methyl-6-(trifluoromethyl)nicotinamides, ethyl 5-salicyloyl-2-methyl-6-(trifluoromethyl)nicotinate, and ethyl 5-hydroxy-2-methyl-5-(polyfluoroalkyl)-5H-chromeno[4,3-b]pyridine-3-carboxylates



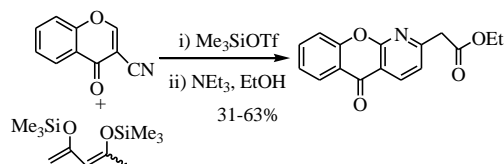
Scheme 17

. Thakur *et al.* explained [18] 4-biradicals generation in the Norrish type-II reactions of 3-propargyloxy-2-arylchromones lead to cyclic products involving the 2-aryl group. The formation and distribution of products varied with the nature of the 2-aryl group



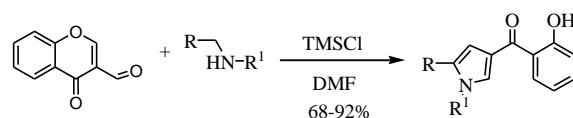
Scheme 18

Rashid *et al.* prepared [19] functionalized 1-azaxanthenes (5-oxo-5H-[1]-benzopyrano[2,3-b]pyridines) by TMSOTf mediated condensation of 1,3-bis(trimethylsilyloxy)-1,3-butadienes with 3-cyanochromones and subsequent base-mediated domino 'retro-Michael/nitrile-addition/hetero-cyclization' reaction.



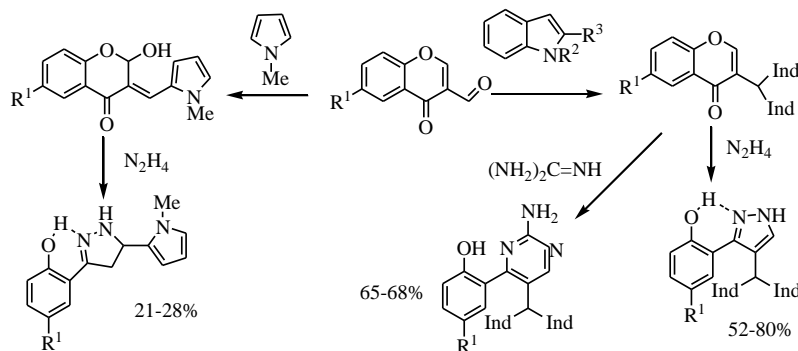
Scheme 19

Plaskon *et al.* established [20] a facile and versatile procedure for the synthesis of 5-hetaryl-3-(2-hydroxybenzoyl)-1H-pyrroles and 4-(2-hydroxybenzoyl)-1H-pyrrole-2-carboxylic acid derivatives on the basis of TMSCl promoted recyclization of 3-formyl-chromone with various hetarylmethylamines and glycine derivatives



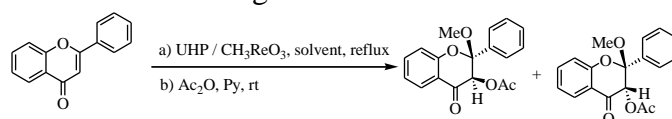
Scheme 20

Sosnovskikh *et al.* synthesized [21](Chromon-3-yl)bis(indol-3-yl)methanes and *E*-2-hydroxy-3-(1-methylpyrrol-2-ylmethylene)chroman-4-ones have been obtained in good yields from 3-formyl-chromones on reaction with indoles and *N*-methylpyrrole under solvent-free conditions. Reactions of (chromon-3-yl)bis(indol-3-yl)methanes with guanidine carbonate and hydrazine hydrate proceed with the participation of the chromone ring system and led to the formation of the corresponding pyrimidines and pyrazoles bearing the bis(indol-3-yl)-methyl moiety.



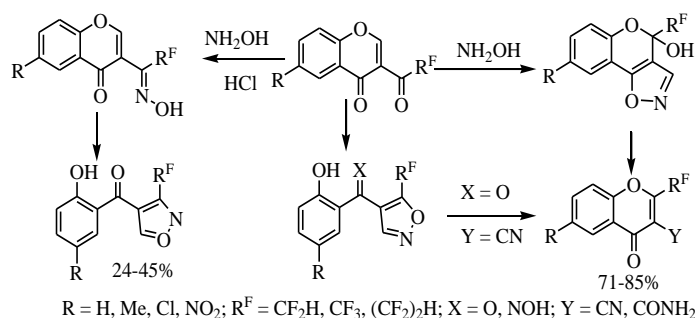
Scheme 21

Bernini *et al.* prepared [22] new *cis*- and *trans*-3-acetoxy-2-methoxyflavanones through epoxidation–methanolysis of monosubstituted flavones with urea–hydrogen peroxide (UHP)/methyltrioxorhenium (CH_3ReO_3 , MTO) catalytic system in methanol as nucleophilic solvent. Some of these compounds are reported to inhibit totally the mycelial growth of some common saprotrophic soil and seed fungi.



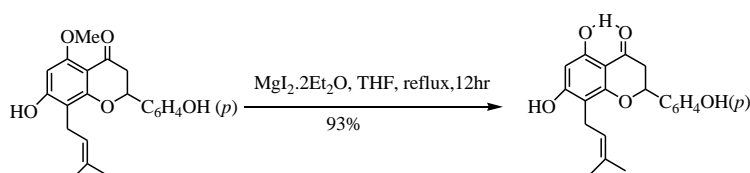
Scheme 22

Sosnovskikh *et al.* explained [23] the reaction of 3-(polyfluoroacyl)chromones with hydroxylamine free base proceeded *via* nucleophilic 1,4-addition followed by opening of the pyrone ring and subsequent cyclization to 4-(polyfluoroalkyl)-4-*H*-chromeno[3,4-*d*]isoxazol-4-ols in good yields. On treatment with trifluoroacetic acid, the isoxazole ring of this annulated heterocyclic system opened to give 3-cyano-2-(polyfluoroalkyl)-chromones, which were successfully hydrolyzed with concentrated H_2SO_4 to afford 3-carbamoyl-2-(polyfluoroalkyl)chromones. On the other hand, oximation of 3-(polyfluoroacyl)chromones with hydroxylamine occurred either at the carbonyl carbon atom connected to the R^{F} group or at the C-2 atom to give 3- $\text{R}^{\text{F}}\text{C}(\text{=NOH})$ -chromones and 5- R^{F} -4-salicyloylisoxazole oximes, respectively. The former were easily converted to 3- R^{F} -4-salicyloylisoxazoles by simple heating in dimethyl sulfoxide



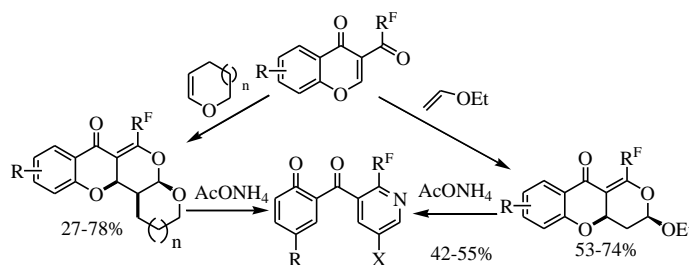
Scheme 23

Anioł *et al.* isolated [24] xanthohumo from supercritical carbon dioxide–spent hop and transformed into isoxanthohumol. The demethylation of isoxanthohumol with the best yield 93% occurred when MgI_2 etherate in anhydrous THF was applied.

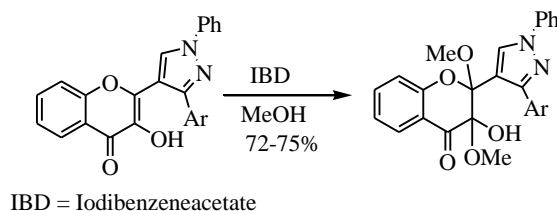


Scheme 24

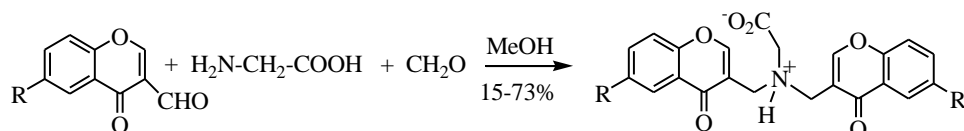
Sosnovskikh *et al.* Explained [25] heterodyne cycloaddition reaction of 3-(Polyfluoroacyl)chromones to 3,4-dihydro-2H-pyran, 2,3-dihydrofuran and ethyl vinyl ether under mild conditions, producing novel fused pyrans with high stereoselectivity and in good yields. Some of these pyrans were transformed into 2-R^F-containing pyridines on treatment with ammonium acetate in ethanol.

**Scheme 25**

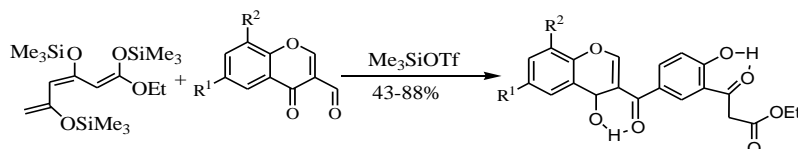
New 2,3-dimethoxy-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl)chromanones have been synthesized by Prakash *et al.* through the oxidation of 3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl)chromones with iodobenzene diacetate in methanol [26].

**Scheme 26**

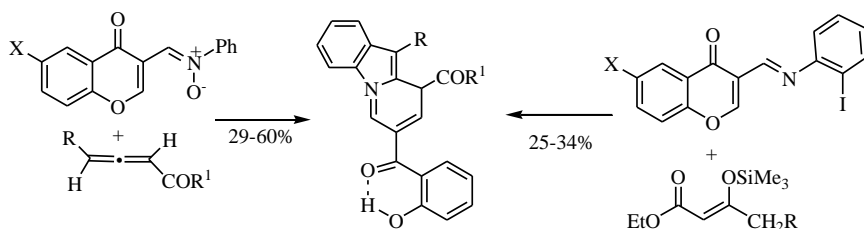
Panja *et al.* reported [27] the reaction of chromone-3-carbaldehydes with N-methylglycine or glycine in the presence of excess formaldehyde to produce N-(chromone-3-ylmethyl)-N-methylglycine or N,N-di(chromone-3-ylmethyl)glycine, respectively, by a deformylative Mannich type reaction.

**Scheme 27**

Adeel *et al.* prepared [28] highly functionalized polyketide-type phenols by domino 'Michael/retro-Michael/aldol' reactions of 3-formylchromones with 1-ethoxy-1,3,5-tris(trimethylsilyloxy)-1,3,5-hexatriene and its synthetic precursor, ethyl 3,5-bis(trimethylsilyloxy)-2,4-hexadienoate.

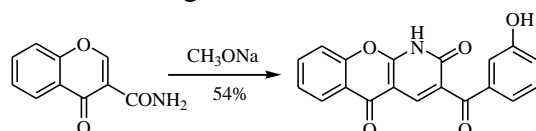
**Scheme 28**

Kapur *et al.* explained [29] the reactions of (4-oxo-4H[1]benzopyran-3-yl)-N-phenylnitrones with allenic esters and allenic ketones furnished benzoindolizines in good yields. The formation of benzoindolizines was postulated to involve regioselective addition of 1,3-dipole to C₂-C₃ pi-bond of allenic esters/ketones followed by domino transformation of the cycloadducts, which involve an intramolecular aza Diels-Alder reaction in the intermediate.



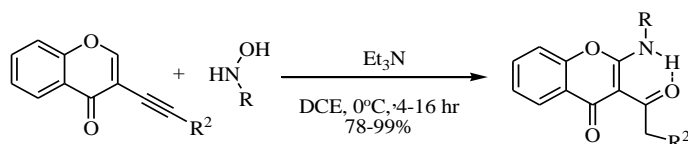
Scheme 29

Ibrahim *et al.* synthesized [30] 3-(2-hydroxybenzoyl)-2H-chromeno[2,3-*b*]pyridine-2,5(1*H*)-dione on treatment of chromone-3-carboxamide with sodium methoxide gave



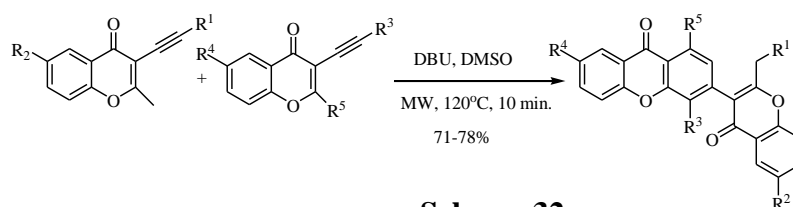
Scheme 30

Zhang *et al.* developed [31] Et₃N-catalyzed tandem reaction of electron-deficient 1,3-conjugated enynes with hydroxylamines was which provided rapid, metal-free, and regioselective access to highly substituted multifunctionalized 2,3-dihydrisoxazoles under mild conditions. The reactions of 3-(2-arylethynyl)-4*H*-chromen-4-ones with hydroxylamines afforded amino enones under the same reaction conditions.



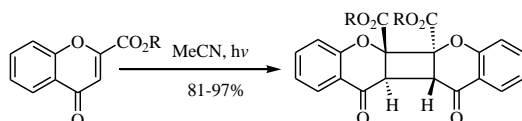
Scheme 31

Gong *et al.* developed [32] a novel base-promoted cascade reaction of 2-methyl-3-(1-alkynyl)chromones which produce a 3*C*-xanthone-linked 3*C*-chromone. This tandem process involves multiple reactions such as Michael additions/cyclizations under mild conditions without a transition metal catalyst in an inert atmosphere



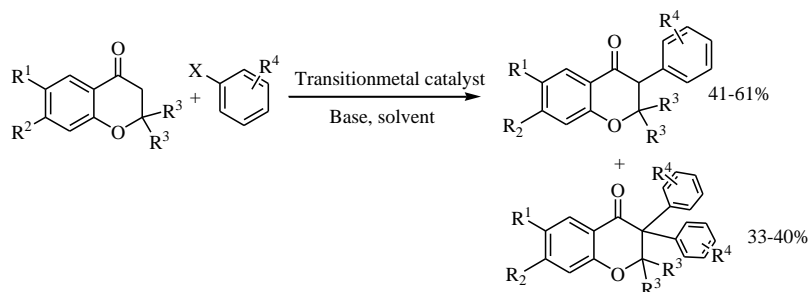
Scheme 32

Sakamoto *et al.* explained [33] photoirradiation of chromone-2-carboxylic esters resulted in the stereo- and regioselective formation of *C*-2 chiral *anti*-HH dimers from the triplet excited state. On the contrary, photolysis in the solid-state gave *anti*-HT dimers exclusively controlled by molecular arrangement in the crystal

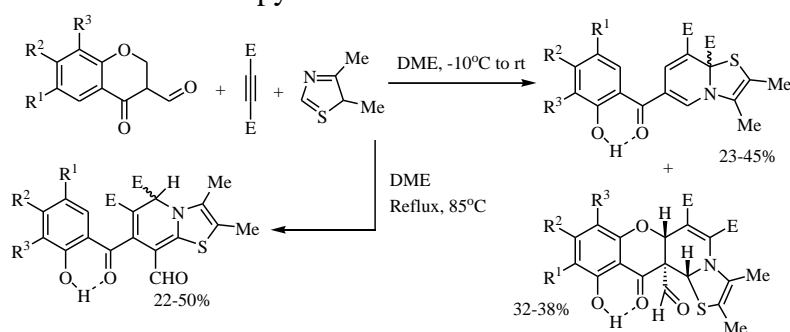


Scheme 33

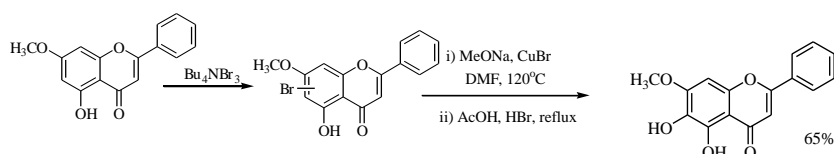
Bellina *et al.* explored [34] the synthesis of racemic isoflavanones in satisfactory to good yields and with high selectivity by Pd-catalyzed direct *C*-3 arylation of 3-unsubstituted 4-chromanones with aryl bromides with the aid of a Pd₂(dba)₃/tBu₃PHBF₄ catalyst system in the presence of KHCO₃ as the base in a dioxane/water mixture (4:1)

**Scheme 34**

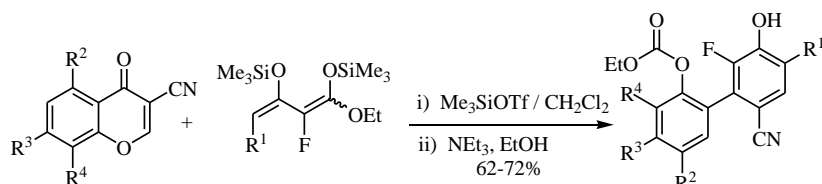
Terzidis *et al.* explained [35] the reaction between 1, 4-zwitterion derived from (4,5-dimethylthiazole and acetylene dicarboxylates) and formylchromones (chromone-3-carboxaldehydes) at low temperature resulting, thiazolo[3,2-*a*]pyridine derivatives after an unusual rearrangement. In the case of electron-donating substituents in the chromone ring tetracyclic chromenothiazolopyridines were isolated as the main reaction products.

**Scheme 35**

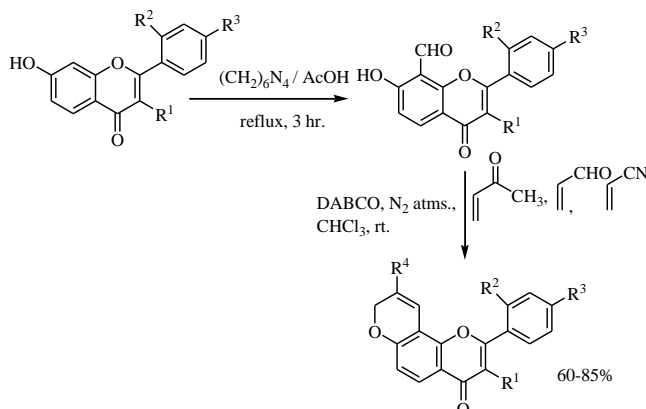
Righi *et al.* developed [36] base induced Wesley–Moser type rearrangement to produce dihydroxy flavanone derivative from monohydroxy flavanone derivative.

**Scheme 36**

Ibad *et al.* explained [37] the Me₃SiOTf-mediated condensation of 1-ethoxy-2-fluoro-1,3-bis(trimethylsilyl-oxy) 1,3-dienes with 3-cyanochromones afforded 3-cyano-2-(4-ethoxy-3-fluoro-2,4-dioxobutyl)-chroman-4-ones. Their reaction with triethylamine afforded fluorinated azaxanthenes or biaryls. The product distribution depends on the structure of the diene. The formation of the biaryls can be explained by an unprecedented domino “retro-Michael/aldol/fragmentation” reaction.

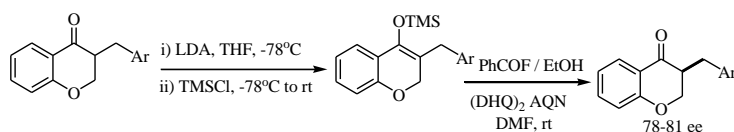
**Scheme 37**

Reddy *et al.* developed [38] the synthesis of 9-acetyl / formyl / cyano-substituted pyrano 2,3-*f*]flavones and chromones through condensation of 8-formyl-7-hydroxyflavones and 8-formyl-7-hydroxy-2-(2'-furyl)-3-methylchromone with methyl vinyl ketone, acrolein, and acrylonitrile in the presence of diazabicyclo[2.2.2]octane (DABCO) under an N₂ atmosphere at room temperature using Baylis–Hillman reaction conditions.



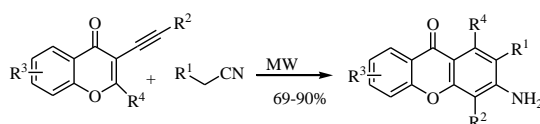
Scheme 38

Poisson *et al.* reported [39] enantioselective protonation of silyl enol ethers by means of a variety of chiral nitrogen bases as catalysts, mainly derived from cinchona alkaloids, in the presence of various protic nucleophiles as proton source.



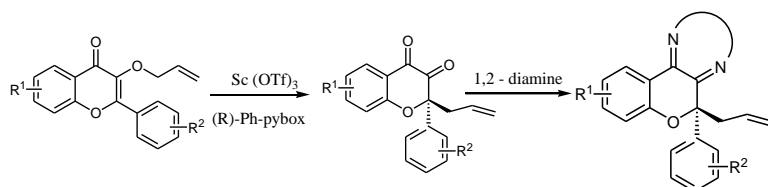
Scheme 39

Liu *et al.* explored [40] One-Pot Tandem Reaction of 3-(1-alkynyl)chromones under Microwave irradiation to Functionalized Amino-Substituted Xanthenes.



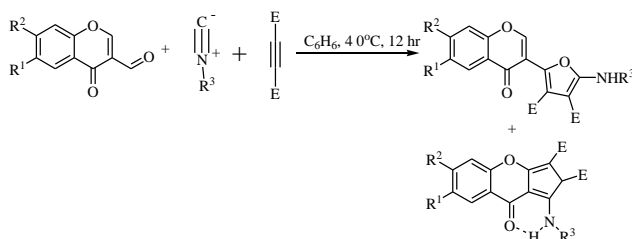
Scheme 40

Xiong *et al.* synthesized [41] chiral, nonracemic 3,4-chromanediones in high yields and enantioselectivities using symmetric scandium(III)-catalyzed rearrangement of 3-allyloxyflavones. These synthetic intermediates were further elaborated to novel heterocyclic frameworks including angular pyrazines and dihydro-pyrazines. The absolute configuration of rearrangement products was initially determined by a nonempirical analysis of circular dichroism (CD) using time-dependent density functional theory (TDDFT) calculations and verified by X-ray crystallography of a hydrazone derivative. Initial studies of the mechanism support an intramolecular rearrangement pathway that may proceed through a benzopyrylium intermediate



Scheme 41

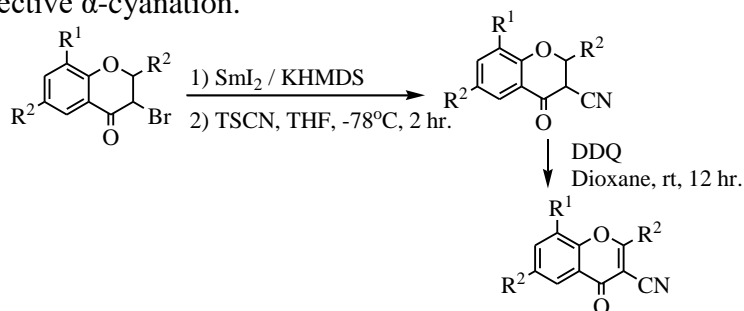
Terzidis *et al.* explored [42] multicomponent reaction leading to 3-(2-furyl)chromones and 2,3-cyclopentadine fused chromones



Scheme 42

Ankar *et al.* utilized [43] a novel combination of SmI₂, KHMDS, and TsCN to introduce a cyano group into structurally diverse and highly sensitive 2-alkyl-chroman-4-ones. Subsequent oxidation allows the formed 2-alkyl-3-

cyanochromones to be isolated in yields ranging from 49 to 77%. In addition, α -bromoketones and esters were found to undergo equally effective α -cyanation.



Scheme 43

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